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TITLE: Automated sampling methods for rapid characterization of polymers

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PARENT-CASE:

This application claims priority under 35 U.S.C. Sec. 119(e) to U.S.

Provisional Application Ser. No. 60/080,652, filed Apr.

3, 1998 by Safir et

al., which is hereby incorporated by reference for all purposes. This

application is related to following U.S. patent applications filed on the date

even herewith, each of which is hereby incorporated by reference for all

purposes: Ser. No. 09/285,393, entitled "Rapid Characterization of Polymers",

filed Apr. 2, 1999 by Safir et al. under; Ser. No.

09/285,333, entitled

"High-Temperature Characterization of Polymers", filed Apr. 2, 1999 by Petro

et al. under; Ser. No. 09/285,335, entitled

"Flow-Injection Analysis and

Variable-Flow Light Scattering Apparatus and Methods for Characterizing

Polymers", filed Apr. 2, 1999 by Nielsen et al. under; and Ser. No.

09/285,392, entitled "Indirect Calibration of Polymer Characterization

Systems", filed Apr. 2, 1999 by Petro et al. under.

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Brief Summary Text - BSTX:

Liquid chromatography is well known in the art for characterizing a polymer sample. Liquid chromatographic techniques employ separation of one or more components of a polymer sample from other components thereof by flow through a chromatographic column, followed by detection of the separated components with a flow-through detector. Approaches for liquid chromatography can vary, however, with respect to the basis of separation and with respect to the basis of detection. Gel permeation chromatography (GPC), a well-known form of size exclusion chromatography (SEC), is a frequently-employed chromatographic technique for polymer size determination. In GPC, the polymer sample is separated into components according to the hydrodynamic volume occupied by each component in solution. More specifically, a polymer sample is injected into a mobile phase of a liquid chromatography system and is passed through one or more chromatographic columns packed with porous beads. Molecules with relatively small hydrodynamic volumes diffuse into the pores of the beads and remain therein for longer periods, and therefore exit the column after molecules with relatively larger hydrodynamic volume. Hence, GPC can characterize one or more separated components of the polymer sample with respect to its effective hydrodynamic radius (R_{sub.h}). Another chromatographic separation approach is illustrated by U.S. Pat. No. 5,334,310 to Frechet et al. and involves the use of a porous monolithic stationary-phase as a separation medium within the chromatographic column, combined with a mobile-phase composition gradient. (See also, Petro et al, Molded Monolithic Rod of Macroporous Poly(styrene-co-divinylbenzene) as a

Separation Medium for
HPLC Synthetic Polymers: "On-Column"
Precipitation-Redisolution Chromatography
as an Alternative to Size Exclusion Chromatography of
Styrene Oligomers and
Polymers, Anal. Chem., 68, 315-321 (1996); and Petro et al,
Immobilization of
Trypsin onto "Molded" Macroporous Poly (Glycidyl
Methacrylate-co-Ethylene
Dimethacrylate) Rods and Use of the Conjugates as
Bioreactors and for Affinity
Chromatography, Biotechnology and Bioengineering, Vol. 49,
pp. 355-363
(1996)). Chromatography involving the porous monolith is
reportedly based on a
precipitation/redisolution phenomenon that separates the
polymer according to
size--with the precipitated polymer molecules selectively
redisolving as the
solvent composition is varied. The monolith provides the
surface area and
permeation properties needed for proper separation. Other
separation
approaches are also known in the art, including for
example, normal-phase
adsorption chromatography (with separation of polymer
components being based on
preferential adsorption between interactive functionalities
of repeating units
and an adsorbing stationary-phase) and reverse-phase
chromatography (with
separation of polymer components being based on hydrophobic
interactions
between a polymer and a non-polar stationary-phase). After
separation, a
detector can measure a property of the polymer or of a
polymer component--from
which one or more characterizing properties, such as
molecular weight can be
determined as a function of time. Specifically, a number
of molecular-weight
related parameters can be determined, including for
example: the weight-average
molecular weight ($M_{\text{sub}.\text{w}}$), the number-average molecular
weight ($M_{\text{sub}.\text{n}}$), the
molecular-weight distribution shape, and an index of the
breadth of the
molecular-weight distribution ($M_{\text{sub}.\text{w}} / M_{\text{sub}.\text{n}}$), known as

the polydispersity index (PDI). Other characterizing properties, such as mass, particle size, composition or conversion can likewise be determined.